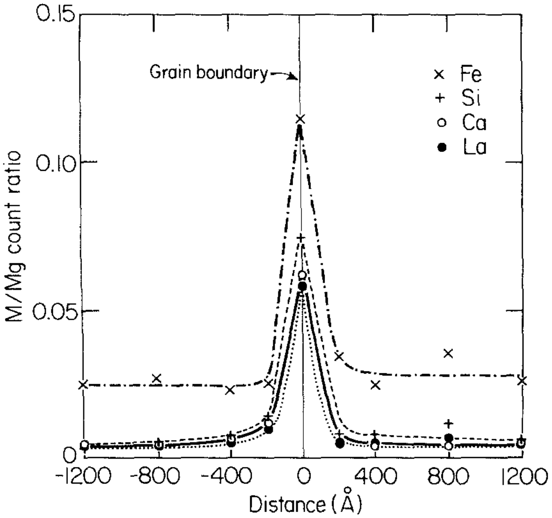

Figure 2 Results of X-ray microanalysis across boundary shown in Fig. 1. Results are given in X-ray count ratios versus position.

lanthanum (this work), it seems clear that segregation of solutes at grain boundaries in ionic oxides such as MgO is very common indeed, and that this factor must generally be taken into account in discussing properties and behaviour of this class of materials.

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Factors affecting the stress dependence for creep of polycrystalline magnesia

During high temperature creep of polycrystalline ceramics, the variation of the secondary creep rate, $\dot{\epsilon}_s$, with applied stress, σ, at a constant temperature, T, can be expressed as

$$\dot{\epsilon}_{s|T} \propto \sigma^n.$$

When creep occurs by the generation and movement of dislocations the stress exponent, n, is usually found to be within the range 3 to 5. Indeed, in the dislocation creep regime, it has been proposed [1] that ceramics can be classifed into two groups depending on whether the stress exponent is approximately 5 (e.g. NaCl, LiF, UO_2) or close to 3 (e.g. MgO, BeO, Al_2O_3). Alternatively, it has been suggested that a specific n value of 3 or 5 should not be assigned to each ceramic [2] since evidence is available which indicates that the stress exponent can depend on the fabrication procedures used to produce the material [3]. In the present work, a study has been made of the effects of changes in the method of sample preparation on the stress exponent for creep of polycrystalline MgO.

Specially prepared $Mg(OH)_2$ was calcined at either 1273, 1373 or 1473 K. The resulting material was then pressed into bars (6.4 mm x 6.4 mm x 32 mm) and sintered at 2073 K in air. The detailed analysis (wt%) was therefore the same for all samples, namely, 99.85% MgO, 0.1% SiO_2, 0.02% CaO, Al_2O_3 and Fe_2O_3 and 0.012% B_2O_3. Moreover, irrespective of the calcination temperature used, samples were produced having 93 to 95% theoretical density and having a uniform grain diameter in the range 10 to 14 μm. Cylindrical testpieces, 4.25 mm diameter and 6.4 mm long, were ultrasonically trepanned from the bars and compression creep tests were carried out over a range of stresses at 1596(± 1) K. The constant

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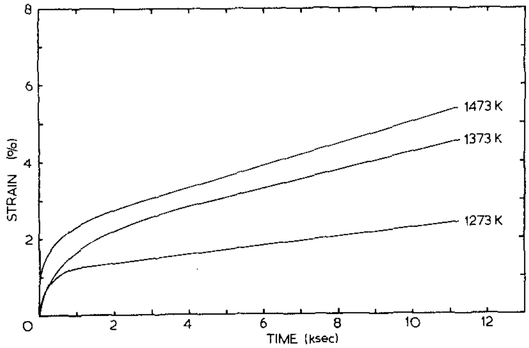

Figure 1 Primary and secondary creep curves recorded at 75 MN m^{-2} and 1596 K for polycrystalline magnesia. The samples examined were fabricated from $Mg(OH)_2$ powder which had been calcined at the temperatures indicated, pressed into bars and then sintered at 2073 K.

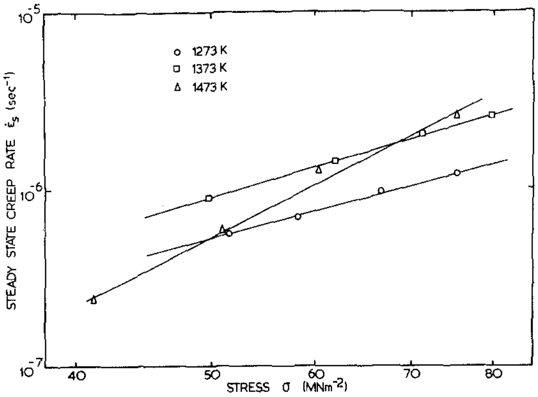

Figure 2 The variation of the secondary creep rate, $\dot{\epsilon}_s$, with applied stress, σ, for tests carried out at 1596 K with polycrystalline magnesia. The samples examined were produced from $Mg(OH)_2$ powder which had been calcined at the temperatures indicated, pressed into bars and then sintered at 2073 K.

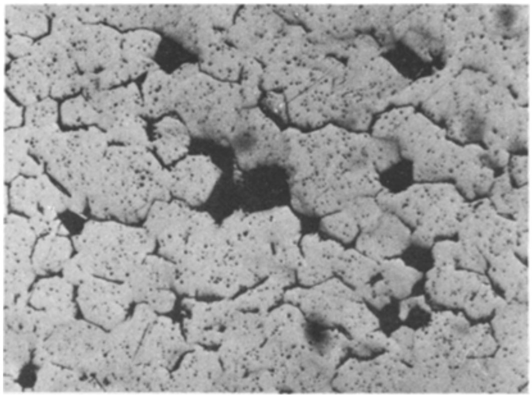

Figure 3 Intergranular cracking evident on transverse section of polycrystalline magnesia sample after a creep strain of 0.05 during a test carried out at 75 MN m^{-2} and 1596 K. This material was fabricated from $Mg(OH)_2$ which had been calcined at 1273 K, pressed into bars and sintered at 2073 K (× 350).

stress equipment, capable of resolving changes in specimen length to 10 nm, has been described previously [4].

For all of the materials studied, the high initial creep rates observed immediately after loading decreased gradually throughout the primary stage until a constant rate was attained during the secondary stage (Fig. 1). Although the general shapes of the creep curves recorded and the creep resistance of the various materials tested were similar for the range of calcination temperatures examined, the stress dependence of the secondary creep rate varied considerably. The n values obtained were 1.8, 2.3 and 3.8 for calcination temperatures of 1273, 1373 and 1473 K respectively (Fig. 2). The present results therefore confirm that the stress exponent for creep of polycrystalline magnesia can be sensitive even to minor variations in fabrication procedure.

Consideration of the creep behaviour in relation to the temperature of the brittle–ductile transition for ceramics has led to the suggestion that the value of the stress exponent is related to the ductility of the material [2]. Substantial plastic flow can occur with polycrystalline ceramics when the von Misés criterion is satisfied. With MgO this requires both $\{1\,1\,0\}\langle 1\,\bar{1}\,0\rangle$ and $\{0\,0\,1\}\langle 1\,\bar{1}\,0\rangle$ slip. The condition that slip takes place at similar stress levels on both $\{1\,1\,0\}$ and $\{0\,0\,1\}$ planes is met only at temperatures above ~ 2000 K [5]. Yet, with magnesia, minor differences in pore size and distribution, composition and grain size arising as a consequence of different fabrication techniques can result in significant variations in mechanical properties, especially in the temperature representing the onset of general ductility [5]. This behaviour has been interpreted on the basis that plastic flow can occur in polycrystalline MgO even at temperatures below ~ 1500 K provided that the grain boundaries are sufficiently strong to allow stress concentrations to build up to initiate slip on the $\{0\,0\,1\}$ $\langle 1\,\bar{1}\,0\rangle$ system [6].

Microstructural examination of the materials prior to testing revealed that the volume fraction and distribution of pores were similar, independent of the calcination temperature used. However, after a given creep strain, the extent to which grain boundary cracks developed during creep was markedly greater for the samples produced using lower calcination temperatures. The extensive cracking evident after a creep strain of 0.05 for the sample which had been calcined at 1273 K is shown in Fig. 3. Intergranular cracks therefore seem to form more easily on boundaries present in samples exhibiting low n values. As concluded from the studies of the plasticity of MgO [5, 6], the detailed fabrication processes used to prepare the samples then appear to affect the stress exponent by determining the ability of grain boundaries to resist cracking. In this way, the present observations are consistent with the view [2] that factors which influence the ductility of polycrystalline ceramics also affect the value of the stress exponent for creep.

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A self-toughening mechanism in epoxide resins

Interest has recently flourished in the fracture behaviour and failure mechanisms in epoxide resins, since these thermosetting polymers are being increasingly employed in structural engineering applications both as adhesives and as matrices in composite materials. A few years ago several papers were published [1, 2] concerning the static fatigue of epoxide resins both in bulk and in adhesive joint specimens and a failure criterion was advanced [2] based upon the attainment of a critical plastic zone size at a crack tip. However, more recently it has been reported [3, 4] that certain epoxide resins of different compositions did not appear to suffer from static fatigue, even when stressed to a relatively high level. This letter outlines further interesting aspects of this observation and demonstrates that an epoxide resin adhesive may even be toughened appreciably by subjecting it to an applied load.

The specimen geometry employed for the adhesive joints was a tapered double cantilever beam joint. The substrate material was aluminium alloy, to specification British Standard 1474 NE4, which was machined into cantilever beams 308 mm long, 12.7 mm thick and with a height, h, varying between 16.0 and 47.8 mm. The surfaces to be bonded were first subjected to a liquid- and vapour-degreasing bath of trichloroethane, then grit blasting with 180–220 mesh alumina; then after degreasing again were finally allowed to dry in air. The epoxide adhesive employed was a diglycidyl ether of bisphenol A cross-linked with 10.0 mass per cent of an amine curing agent (tetraethylenepentamine). Immediately prior to joint preparation the aluminium alloy substrates were treated as described above. Adhesive was spread on the treated faces and the two beams pressed lightly together. Small pieces of plastic

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